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## NANOSTRUCTURED COATINGS OF INNER SURFACES IN MICROPOROUS MATRIXES.

Yu.A. KUMZEROV

*A.F.Ioffe Physico-Technical Institute, Russian Academy of Sciences,  
194021 St.Petersburg, Russia*

### 1. Introduction

There is growing interest in the physical properties of extremely small structures. The experimental realisation of new effects relies on the ability to create new types of structures and devices. Our understanding of material processing in the pursuit of ultra-small structures is continually advancing. Sophisticated epitaxial growth and lateral microstructuring techniques have made it possible to realise low-dimensional electronic systems with quantum confined energy structure i.e. quantum wells, quantum wires and quantum dots. Low-dimensional systems can also be obtained by confining a solid or liquid within the nanometer-sized pores of different porous materials (see [1]). Systems with size-selected nanoparticles embedded in a porous matrix via chemical coating have received some attention these past few years. In this case the confined materials penetrate into the pores due to wetting processes. In the case of non-wetting there is the possibility of using a mechanical coating of inner surfaces of porous materials when some external pressure forces non-wetting liquid into the pores [2]. Mechanical coating means that the mechanical energy of the piston forcing the liquids into the pores transforms to the energy of some new surface arising due to filling process (Fig.1). Since the total surface energy is proportional to the highly developed inner surface of the porous matrix, to surface tension of embedded materials and inversely proportional to the characteristic diameters of pores, for very small diameters a relative amount of surface energy may be very large and even comparable with some traditional sources of energy (Fig.1). For example, liquid mercury embedded into the pores of zeolites (needed pressure is about 20 kbars) accumulates of about 0.4 kJ/g mechanical energy in the form of a surface energy. Such a situation may be used for mechanical energy accumulation [3]. On the other hand the materials coating the inner surfaces of the pores in different porous matrixes may be interesting themselves as some physical objects with the characteristic sizes in nanometre scale i.e. as some new types of nanostructures. The advantages of such nanostructures are connected with the possibility to produce the following:

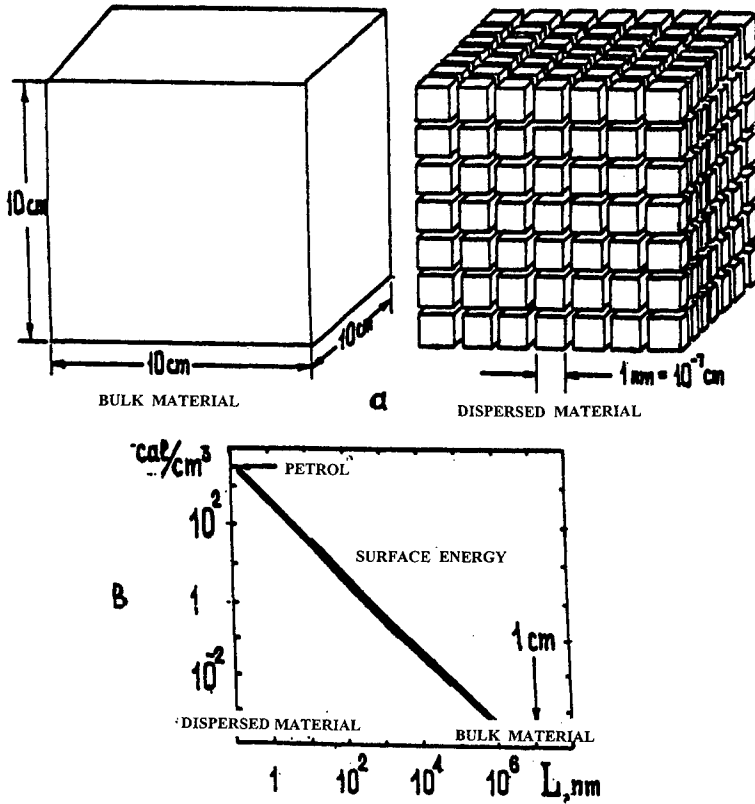


Figure 1. a) Surface arising due to process of material dispersion.

b) Surface energy per  $\text{cm}^3$  of dispersed material versus characteristic size of dispersed particles

- nanostructures with different geometries ( two-dimensional film-like structures, one-dimensional wire-like structures, zero-dimensional small particles-like structures).

- nanostructures with a large range of characteristic sizes ( from approximately 1 nm to approximately 100-150 nm).

- nanostructures with very large total amount of nanomaterial ( up to some cubic cm). Such situations permit the use some experimental methods ( neutron scattering, heat capacity measurements) that require a large amount of nanostructures.

- nanostructures from different substances ( from metals, including superconductors, from semiconductors, from insulators, including ferroelectrics etc). Further, examples of such nanostructures, and their physical properties will be presented.

## 2. Irregular systems of small particles in porous glasses.

Small particles or film-like objects can be obtained by confining a solid or liquid within the nanometer-sized pores of porous glasses. Porous silica glasses have often been used for this purpose because of their relatively well-characterised pore structure, large specific pore volumes and availability over several orders of magnitude in the mean pore diameter (about their structure see [4]). The porous matrix usually is prepared from a sodium borosilicate glass. This glass is produced from a melt of 75%  $\text{SiO}_2$ , 20%  $\text{B}_2\text{O}_3$  and 5%  $\text{Na}_2\text{O}$ . Heat treatment leads to a separation in a silicon-rich phase and a boron-rich one. After removing the boron-rich phase, there remains an interconnected solid skeleton of nearly pure  $\text{SiO}_2$  with a rather homogeneous distribution of mass and pores. The pores have a rather narrow size distribution and the pore diameters may be varied from 4 nm to about 100 nm. The pores together with narrow necks, which connect the pores, form a random interconnected network in a glass bulk.

Phase transitions in materials confined within porous glasses have been studied intensively. The melting and freezing within porous glasses have been observed for materials such as water, organic liquids [5,6], metals with low melting point [7-11], helium [12], oxygen and some other simple liquids [13-15]. The ferroelectric phase transition within porous glass has been observed for  $\text{NaNO}_2$  and  $\text{K}_2\text{HPO}_4$  [16,17]. Superconductivity for some metals confined within porous glass has also been observed [18].

## 3. Regular systems of small particles in opals.

The regular systems of identical nanoparticles may be obtained by confining some substances within the pores of opal. Opal itself consists of identical silica balls with diameters ( $D$ ) ranging from 150 to 350 nm (about its structure, see [19]). The size homogeneity of these spheres allows their assembly in a close three-dimensional lattice, usually with FCC symmetry. Empty voids exist between neighbouring balls which, in turn, form their own regular lattice. There are two types of interpenetrating voids in the opal lattice: eight-fold co-ordinated large voids each connected with eight four-fold co-ordinated small voids. A large void has the form of a truncated tetrahedron with four windows to four large voids. The diameters of spheres inscribed in the larger and smaller voids are  $d_1=0.41D$  and  $d_2=0.23D$  respectively. The diameter of a circle inscribed in the triangular window is  $d_3=0.15D$ . The density of voids in opal is typically  $10^{14}\text{cm}^{-3}$ . The porosity of the ideal package of balls is 26% of the whole volume.

This matrix itself is interesting as a material for so-called photonic crystals, that behave with respect to photons like a dielectric crystal does with respect to electron

waves and demonstrates the photonic gap phenomenon [20]. The lattices of nanosize metal particles may be prepared by impregnation of the opal matrix by molten metal under high hydrostatic pressure conditions [21-25]. The shape of the metal grains is a precise copy of the void configuration since the metal occupies all free matrix volume. Thus, a three-dimensional metal replica of the matrix is formed. According to the matrix structure, each section consisting of two adjacent grains contains a constricted region which may be considered as a weak link i.e. the element of S-c-S (S-superconductor, c- constriction) type. Owing to the matrix these elements are arranged in a crystalline manner and form a macrosystem. The density of weak links in this material is near  $10^{14} \text{ cm}^{-3}$ . Defects of this secondary lattice are the same as those of ordinary atomic crystals, and therefore the samples are polycrystals consisting of crystallites with characteristic size near 0.01 cm [23]. This object demonstrated properties characteristic of the Josephson systems [21, 23, 24]. The oscillating dependence of the critical current magnitude on the external magnetic field for such samples was observed and its example is shown in Fig.2 ( from [23]). Such dependence may be connected with a regular structure of investigated materials.

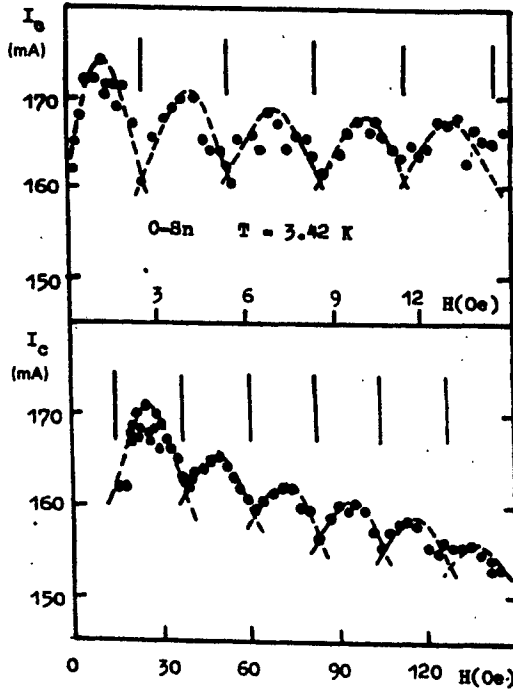


Figure 2. Critical current ( $I_c$ ) vs magnetic field ( $H$ ) for Sn-based sample. The upper plot corresponds to narrow and the lower plot to the wide scale of the magnetic field. The measured points are connected by lines to guide the eye. The sections indicate the periodicity.

#### 4. Nanoclusters in zeolites.

The further type of microporous materials are the crystalline framework solids such as zeolites (about their structure, see [26]), that have found great utility as catalysts and sorption media because of their large internal surface area. They have regular arrays of identical voids, forming three-dimensional channel networks with very small sizes compared with the diameters of the atoms. For example, the channels in NaA zeolite have maximum diameter of 11.4Å, run in three perpendicular directions, and are interrupted by narrow diaphragms having a diameter 4.2Å. The cavities in NaX zeolite form a diamond lattice and have a maximum diameter of 13Å, the diaphragms have a diameter of 8Å.

Wetting liquids such as water will spontaneously fill the channels and cavities of zeolites essentially completely. It was shown in [27,28] that non-wetting liquids (mercury, gallium, bismuth) can be introduced into the regular channels of zeolites at sufficiently high pressures. The pressures at which mercury and gallium fill NaA zeolite are 14.5 kbar and 23 kbar, respectively. The critical pressures for NaX zeolite are 12.5 kbar (Hg) and 20 kbar (Ga). These values can be estimated from well-known porometric equation, based on the concepts of surface tension and wettability of liquid [27]

$$P_c = 0.4\sigma/d,$$

where  $P_c$  is critical pressure (kbar),  $\sigma$  is surface tension (dyn/cm) and  $d$  is the channel diameter (Å). After the external pressure is removed, some of the liquid metal leaves the zeolite channels. Nevertheless, the density of the zeolites shows that about half of the metal which was in the zeolite remains there. The rest of the metal collects in cavities and drops as a result of surface tension thus forming some ordered secondary crystal.

Some of the physical properties of such materials were investigated. For Hg and Ga drops a phase transition was observed [29]. It was manifested by singularities in the temperature dependence in the electrical conductivity and specific heat. The singularities were observed at temperatures much lower than the melting point of the bulk metal. It was concluded that the phase transition occurred in a system of interacting drops composed of metal atoms, and the interaction stabilised the system against fluctuations. The absorption spectra of Se drops in the zeolites NaA and NaX [30] were investigated. The form of the spectra obtained is typical of absorption spectra of crystals in the region of the optical absorption edge. It was concluded that the lattice of the clusters produced in the zeolite cavities is also a crystal, and the obtained spectral curves correspond to the absorption spectra of such cluster crystals in the region of the interband transitions.

A strong nonlinearity of the current-voltage characteristic of the cluster superlattice of tellurium in NaX zeolite was observed [31]. The characteristic was a series of current peaks rising in increasing amplitude in the field. This behaviour is

demonstrated in Fig.3 ( from [31]). It was shown that these peaks appeared periodically when considered as a function of  $1/E$  ( $E$  is electric field intensity). The periodicity of the current peaks ( on the  $1/E$  scale) of the current-voltage characteristic for Te in NaX zeolite was discussed on the basis of a theory of electrophonon resonance.

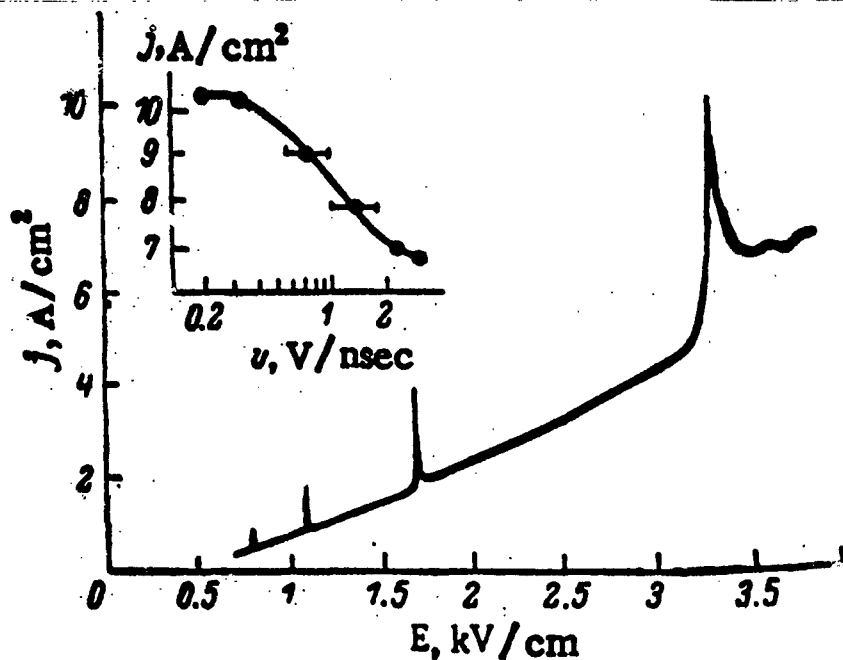


Figure 3. Oscillogram of the current-voltage characteristic obtained using sawtooth voltage pulses with a leading edge  $\tau_f \sim 50$  nsec. The inset at the top of the figure gives the dependence of the amplitude of the  $n=1$  peak on the rate of the rise of the voltage  $v$ .

## 5 Quantum wires in chrysotile asbestos.

Quantum wires of a wide class of materials can be prepared in porous matrices. This is natural chrysotile asbestos mineral ( composition  $Mg_3Si_2O_5(OH)_4$  ). It is a regular set of closely packed parallel ultrathin dielectric tubes with external diameters of  $\approx 30$  nm ( this value determines the distance between the centres of the neighbour channels), and with internal channel diameters of 2-10 nm depending of the origin of the mineral ; its structure is described in [32, 33]. Chrysotile asbestos tubes constitute single-crystal alumino-silicate sheets ( the thickness of one sheet is 7Å), which owing to their structural characteristics twist themselves into rolls until the radius of curvature of the outer and inner layers starts differing appreciably from their optimum value. The tubes of natural chrysotile asbestos reach lengths of several centimetres.

When we fill the channels with molten materials ( Hg, Sn, Bi, In, Pb, InSb, Se, Te) under high pressure conditions we have the possibility of obtaining a regular system of ultrathin parallel wires. Fig.4 demonstrates the model of this system. The reduction in the pressure of the molten metals after their introduction into the asbestos leads to a loss of sample conductivity on account of the oozing of the liquid metals from the channels; it is therefore essential to carry out the conductivity measurements directly under pressure, using the autonomous chamber with a reserve valve.

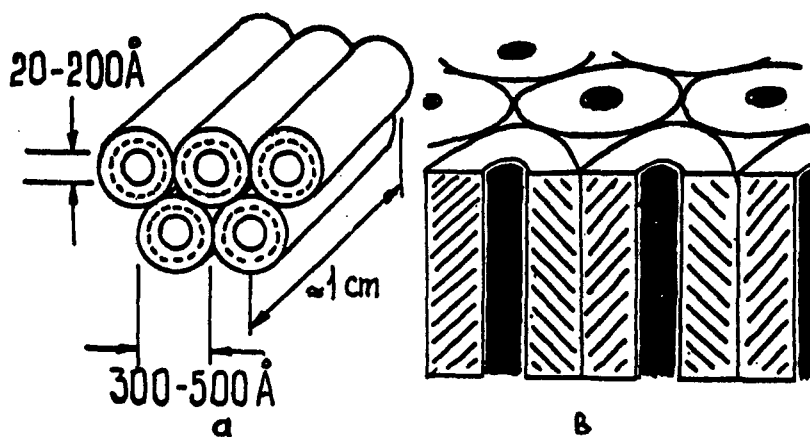


Figure 4. a) Close packed tubes of chrysotile asbestos.  
b) System of ultrathin wires within the channels of chrysotile asbestos

The superconducting transitions of a complete series of such samples by the contact method were studied [34-40]. Particularly, the superconducting transition of metals in asbestos channels has a temperature spread that is due to fluctuations which are significant for such thin elements [35]. Fig.5 demonstrates such behaviour for mercury wires of 80 Å diameter. The measurements performed on mercury wires with diameters 20 Å [35] show that their superconducting transition is smeared from ~2K to ~6K that  $\Delta T/T_c \sim 1$ , where  $\Delta T$  is the width of critical region. Some attempts of theoretical description of the fluctuation smeared superconducting transition are known [41-43] and the theoretical dependence is shown in Fig.5 by the solid line. It may be seen that the theoretical curve in the low temperature region is quite consistent with the experimental data. The critical temperature of the ultrathin wires was determined from resistance dependence on temperature, using some theoretical description of the fluctuation smeared superconducting transition [40]. Critical temperatures vs diameter were measured for mercury, tin and indium with wires from 20 Å to 150 Å. They are demonstrated in Fig.6. The critical temperature of tin wires increases with decreasing diameter and critical temperature on diameter dependence.



for mercury and indium wires have a maximums at 4.6K for diameter  $\approx 40\text{\AA}$  (Hg) and at 6.2K for diameter  $\approx 20\text{\AA}$  (In).

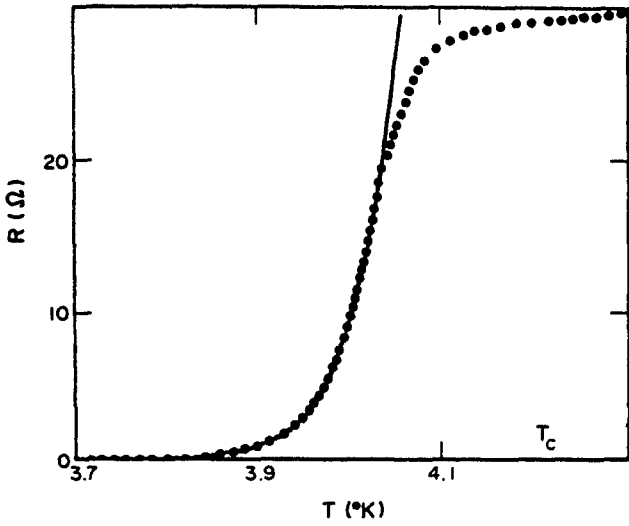


Figure 5. Resistance as a function of temperature for mercury wires of 80 $\text{\AA}$  diameter in the superconducting region

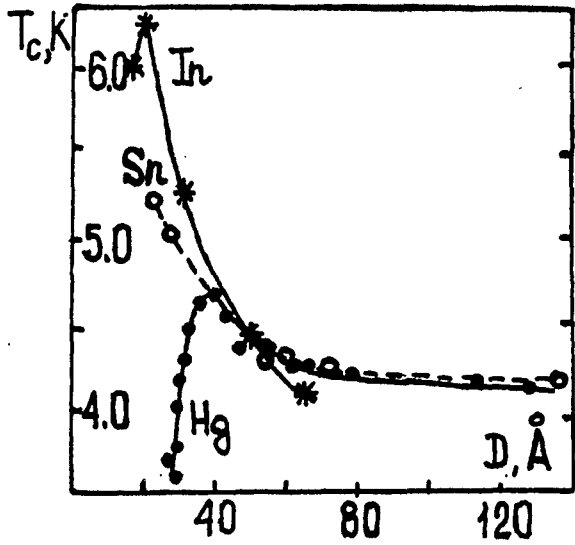


Figure 6. Superconducting critical temperature dependence on the diameter of mercury, tin and indium wires.

Some increase of resistance and non-linearity of current-voltage characteristics with decreasing temperature were observed on mercury ultrathin wires [35,44]. The temperature dependence of mercury 25A wires resistance in the region below 60K is shown in Fig.7. In the temperature range 10-60K the experimental data are well described by  $T^{-3/2}$  dependence. Below 10K the

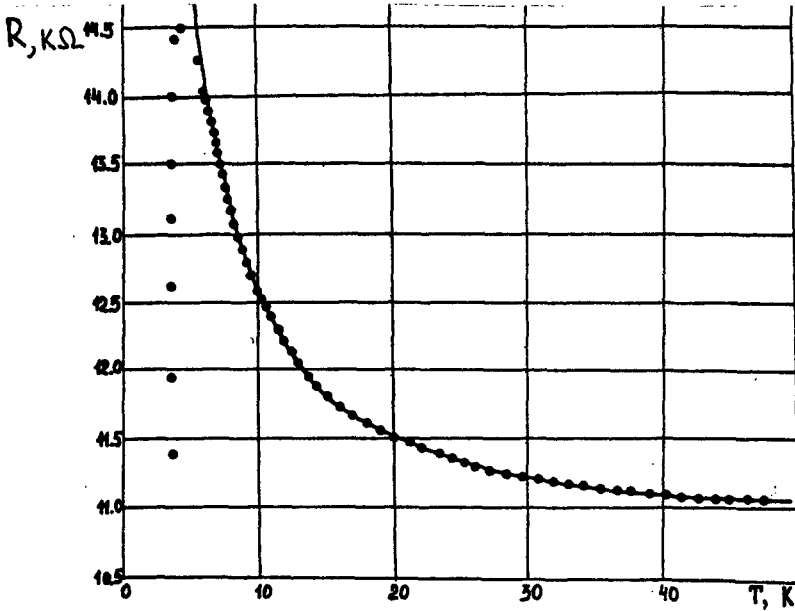


Figure 7. The dependence of resistance of 25A mercury wires on temperature ( the solid line corresponds to  $\Delta R/R \sim T^{-3/2}$  dependence).

superconducting fluctuations dominate the conductivity. As to wires with larger diameters, their negative temperature derivative of resistivity decreases with the diameter. These results were explained as the display of localisation effects in disordered one-dimensional system. The temperature dependence of resistivity of thin wire due to localisation ( one-dimensional case) can be written as [46]

$$R = R_0 [1 + \rho_B (D\tau_i)^{1/2} / R_T d^2]$$

where  $\rho_B$  is the impurity scattering resistivity,  $D = v_F l / 3$  is the diffusion constant,  $v_F$  is the Fermi velocity,  $R_T = 36500 \Omega$ ,  $d$  is diameter of the wire,  $l$  is the elastic mean free path,  $\tau_i$  is the inelastic scattering time. The necessary condition for one-dimensional behaviour is  $d < (D\tau_i)^{1/2}$ . The temperature dependence of wire resistance in this equation is governed by the temperature dependence of  $\tau_i$ , so that  $\Delta R/R_0 = (R - R_0)/R_0 \sim (\tau_i)^{1/2}$ . Since we have  $\Delta R \sim T^{-3/2}$  dependence, it follows that  $\tau_i \sim T^{-3}$ . Such temperature dependence of inelastic scattering time may be due to scattering on three-dimensional

phonons. Using this equation we find  $\tau_i \sim 10^{-10}$  sec at 10K. The localisation length  $(D\tau_i)^{1/2}$  at 10K is about  $3 \times 10^{-5}$  cm which satisfies the localisation criterion for wires and so the data obtained may be interpreted in terms of one-dimensional electron localisation.

The melting and freezing as a first-order phase transition of mercury wires in the range between 20Å and 100Å [45] was investigated. The first-order phase transitions in dispersed systems have been studied considerably less extensively than the second-order phase transitions. There have been virtually no experiments of this type performed using approximately one-dimensional systems as objects of investigation. The melting region of extremely fine mercury wires was identified on the temperature curve of the resistance. It was of considerable interest to study this transition as a function of wire diameter. The samples whose wires had a small spread in diameter (not exceeding  $\sim 5\%$ ) were chosen for measurements. The melting region extends over a large temperature interval, which is probably attributable to the fluctuations whose appreciable effect on the phase transitions in one-dimensional systems has been well established. At such a highly diffuse transition, it is not clear what point should be regarded as the melting point. Since there are no physically sound criteria for determining this point on the resistance versus temperature curve in the melting region (although these criteria can be determined for second-order phase transitions in several cases [40]), we can arbitrarily assume that the melting point is that point at which the resistance changes only half as much as it does during melting.

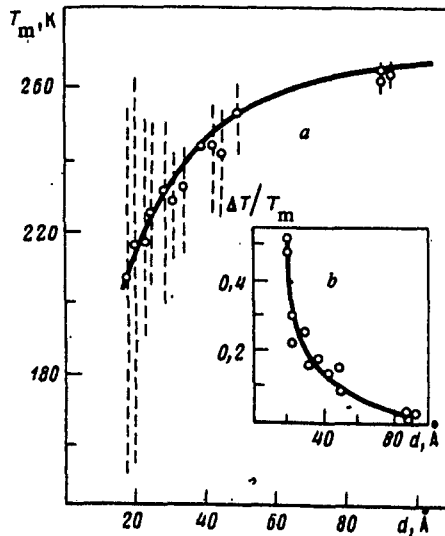


Figure 8. a) The melting point of extremely fine mercury wires versus the diameter (dashed line corresponds to the diffuse-transition region).  
b) The diffusive melting range versus diameter

The dependence of a particular melting point on the diameter is shown in Fig.8. Also shown here is the region in which the transition becomes diffuse (dashed lines). The solid curve corresponds to the dependence  $T=T_0(1-d^*/d)$ , where  $d^*\sim 5.5\text{\AA}$  and  $T_0\sim 280\text{K}$ , which is equal to the melting point of bulk mercury at a pressure of  $\sim 10$  kbar. Fig.2b is a plot of  $\Delta T/T_m$  ( $\Delta T$  is the diffuse-transition region) as a function of the diameter, and the solid curve corresponds to the relation  $\Delta T/T_m=(D/d)^2$ , where  $D\sim 14\text{\AA}$ .

In addition some optical properties of ultrathin wires incorporated within chrysotile asbestos nanotubes were studied. Polarised optical absorption spectra of Hg, Bi and InSb wires have been studied in the spectral region 0.5-3 eV, and high anisotropy of the absorption has been observed [47,48]. InP quantum wires have been produced in channels of asbestos by metal-organic chemical vapour deposition [49]. A comparative study of Raman, optical absorption, and photoluminescence spectra revealed the dependence of the optical properties of quantum wires on interface effects, namely, atomic interaction in the wires, wire-matrix, and wire-wire interactions.

## 6. Conclusions.

The results obtained clearly show good potential for the use of the natural and artificial microporous media for the preparation of the artificial systems that are interesting for scientific investigations and potential applications. The utilisation of porous glasses, artificial opals, and chrysotile asbestos matrices allows the formation of nanostructures for measurement of electrical, optical and other properties.

## 7. Acknowledgements.

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